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in Gallium Single Crystals

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S. H. McFarlane III and C. Elbaum

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S. H. McFarlane III and C. Elbaum

Metals Research Laboratory

Department of Physics

Brown University

Providence, Rhode Island

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Abstract

The development of dislocation networks in gallium crystals, initially free of detectable dislocations, has been studied at room temperature by an x-ray diffraction topographic technique. The condition required for the development of these networks is the existence of spatial periodic fluctuations in impurity content. The networks develop, in the absence of an external stress, from individual loops which are first detected 3 to 10 days after the crystal has been grown. These loops grow for days at a uniform rate of 10^{-8} cm/sec and reach a size of a few hundred microns. No specific nucleation mechanism for these loops has been identified. If it is assumed that the loops expand by climb (by the addition of vacancies), their rate of growth and observed densities are consistent with a formation energy for vacancies U_f in the range 0.2 to 0.3 ev and a migration energy for vacancies U_m in the range 0.4 to 0.7 ev. No values of U_f, U_m or $U_f + U_m$ determined from other experiments have been reported and a comparison is not possible at the present time. The change of initially straight dislocation lines into jagged lines has been observed and is interpreted as the climb of screw dislocations into irregular helical dislocations. A marked anistropy in the growth of the loops and helices was observed and is attributed to an anisotropic coefficient of self-diffusion.

Introduction

The origin of dislocations and their development into networks in crystals has been the object of much research and some controversy.[1] In this paper observations are reported on the development of dislocation networks in gallium single crystals grown from the melt. It is shown that crystals essentially free of dislocations detectable by x-ray diffraction topography can be grown consistently by the technique described here. Of even greater interest is the observation that in crystals which contain spatial fluctuations in impurity content dislocation networks develop in the absence of external stresses at room temperature (i.e., within a few degrees of the melting temperature) over periods of many days following solidification.

Experiment

Most of the crystals were grown from gallium nominally of 99.999% purity supplied by AIAG Metals, Inc. An as-received ingot had a resistivity ratio[2] of $\rho_{273°K}/\rho_{4.2°K}$ = 8,000. A few of the crystals were grown from AIAG gallium nominally of 99.9999% purity which had a resistivity ratio[3] of $\rho_{273°K}/\rho_{4.2°K}$ = 35,000.

The crystals were grown by an unconstrained solidification technique, similar to that used by Kapitza[4] to grow crystals of bismuth. First, a crystal of gallium (2 mm x 0.5 mm x 40 mm) of a desired orientation is grown in a demountable mold. The crystal is removed from the mold and placed on a Teflon surface: either a Teflon block or an acryllic plastic block covered with Teflon tape. One end of the block is placed on a hot plate and warmed just enough to melt the gallium. The shape of the crystal remains essentially

unchanged during the melting if the temperature of the block is kept to within a few degrees of the melting temperature of gallium, 29.8°C. The block is removed from the hot plate before the crystal is melted completely and the loss of heat to the cooler portion of the block and the surroundings provides a sufficient heat-sink for solidification to occur. The adherence of the gallium to the Teflon is the only constraint on the crystal during growth which proceeds at a rate of from 1 to 2 mm/min. The solid-liquid interface can usually be identified as consisting of (111) planes and takes on a well defined pointed shape for the crystal orientations used. A more complete description of the growth procedure has been given elsewhere.[5]

The crystals were examined by the anomalous transmission method of x-ray diffraction topography.[6] The crystal structure of gallium is one-face-centered orthorhombic with 4 atoms per lattice point.[7] Of all the planes with a non-zero structure factor, the (020) set is the only one which will support appreciable anomalous transmission. This is because the (020) planes are the only ones for which all the atoms in the crystal lie in the crystallographic planes. For all other planes the atoms lie above and below the crystallographic planes and attenuate the standing wave which is responsible for anomalous transmission.[8] Hence, for convenience in x-ray observation, the crystals are grown so that the (020) planes are perpendicular to the surface and parallel to the length of the crystal. The availability of only one set of diffracting planes prevents the determination of the Burgers vectors of the dislocations observed by a comparison of dislocation images in topographs from various reflections. The x-ray topographs were taken on Ilford nuclear emulsion G5, 50 μ thick, with the $K\alpha_1$ line of Mo. The crystal

and film were translated back and forth in the diffracted beam during the exposure.[9]

Results and Discussion

Most of the crystals grown were essentially free of dislocations visible in the x-ray topographs. The average dislocation density of the thirty best crystals (out of forty examined) was 30 lines/cm^2. The dislocations which did occur were quite often bunched together so it was not unusual to obtain crystal volumes of 10 to 20 mm^3 completely free of detectable dislocations. It should be emphasized that this is the first reported case of the observation of large volumes of dislocation-free material obtained consistently in a metal crystal grown from the melt. It is also worth emphasizing that the term "dislocation-free" means that the crystal is free of dislocations detectable by x-ray topographic methods. The crystals may contain dislocation loops too small to be resol by x-ray topography.

A few of the crystals exhibited a "banding" structure which is visible in Fig. 1a as parallel regions of contrasting intensity running diagonally across the crystal. The orientation of the bands is consistent with their being parallel to the (111) planes, i.e., parallel to the solid-liquid interface during growth. The line which marks the intersection of the two regions of oppositely slanted bands in Fig. 1a moves from side to side across the crystal in much the same way as the vertex of the pointed solid-liquid interface was observed to move from side to side as the crystal solidified.

Tha banding is attributed to periodic changes in impurity content and may be associated with temperature fluctuations due to thermal convection in the melt during growth. Impurity bands of this type have been observed, for example, in

germanium,[10] silicon,[11,12] and InSb.[13,14] (In the case of silicon, the impurity has been identified[11,12] as oxygen and in InSb the bands have been correlated[13,14] with observed temperature fluctuations of from 1 to 2 °C during growth.) No attempt was made to determine whether temperature fluctuations occurred in the melt from which the banded gallium crystals were grown.

Several observations which support the conclusion that the banding is caused by impurity concentrations are discussed below. A topograph of a crystal grown from two specimens of different impurity content exhibits much stronger anomalous transmission through the region grown from as-received material than through the region grown from contaminated material. The uniform decrease in intensity obtained through the part of crystal of known moderate impurity content is similar to the periodic reduction in intensity of the anomalous transmission which makes the bands visible on the topographs.

All but one of the crystals which exhibited banding were grown from one particular gallium melt. This melt was originally of 99.999% purity but contained remelted crystals which had been grown previously. Semi-quantitative spectrographic analysis[15] showed that the melt had a higher impurity content than the as-received material. Crystals grown from other melts with similar history and impurity content (as detected by spectrographic analysis) did not exhibit banding. The occurrence of banding is not due to different growth conditions as crystals from different melts grown side-by-side do or do not exhibit banding, depending on their parent melt. Banded crystals which are regrown still exhibit banding.

In summary, no crystals grown from as-received material exhibit banding. Crystals grown from some moderately contaminated melts exhibit reduced anomalous

transmission and no banding. Crystals grown from one particular moderately contaminated melt exhibit banding. A crystal grown from a highly contaminated melt exhibited an observable diffracted x-ray intensity only along its edge, i.e., only at the thinnest section (<0.1 mm) of the specimen. Banding was observed in this region. Although the relative purities of the various melts have been determined by semi-quantitative spectrographic analysis, no conclusions can be drawn from the analysis as to what impurities in what concentrations are required for a crystal to exhibit banding when grown under the conditions used in this study.

The crystals which exhibited banding were usually free of detectable dislocations when first examined. In 3 to 10 days dislocation loops would appear and over a period of several days develop into a tangled network of dislocations. (Network development has been observed over a period as long as eight weeks.) This network development was observed only in those crystals which exhibited banding. No banded crystal which was observed for a period of over 10 days failed to develop dislocation loops in regions previously free of detectable dislocations.

The sequence of topographs in Fig. 1 shows several stages in the development of the network structure. Figure 1a shows the crystal one day after it was grown; no dislocations are visible. Figure 1b shows the same crystal 12 days after growth and a number of loops have appeared. Figure 1c shows the crystal 20 days after growth and loops visible in Fig. 1b have increased in size and additional loops have appeared.

The banding in the crystal shown in Fig. 2 was not as pronounced as in any of the other crystals observed and the network development was not as

extensive. The topographs shown were taken at 2,6,13,19,27 and 32 days after growth. The increase in the number of loops as well as the growth of individual loops is very apparent. A set of stereo topographs (not shown here), taken in the manner described by Haruta[16] and by Young et al.,[17] reveals that the loops in the crystal shown in Fig. 2 are distributed throughout the bulk of the crystal. Note that the expansion of the loops in both Figs. 1 and 2 is anisotropic, the greater expansion being in the vertical direction on the topograph.

The fact that the network development occurs only in crystals which exhibit the banding structure indicates that the existence of the loops is related to the distribution of impurities in the crystal. However, no specific mechanism for the nucleation of the loops has been identified. It is possible, of course, that only the growth of the loops is related to the impurity distribution in the crystal and that nucleated loops are present in all of the crystals grown but are too small to be visible on the x-ray topographs.

The rate of growth of several of the loops shown in Fig. 2, as well as in another crystal not shown here, was determined by measuring the length of the loops in successive topographs. The loops all had a constant growth velocity of 10^{-8} cm/sec. This value is based on a measurement of the projection of the loop on the film. If the plane of the loop is not parallel to the film, the actual rate of growth will be greater than this measured value. However, the set of stereo pictures mentioned above indicates that the plane of the loops is nearly parallel to the film, so the velocity measured should be close to the actual rate of growth of the loop.

Since the dislocation networks develop in the absence of an external stress on the crystal, the loops apparently expand by a climb process. If it is assumed that the loops expand by the addition of vacancies, the velocity of climb at a temperature T is given by[18]

$$v = \frac{e^{-U_j/kT}\, e^{-U_m/kT}}{b}\; e^{-U_f/kT_A} - e^{-U_f/kT} \qquad (1)$$

$$= \frac{c_j D_v}{b} (c_A - c_o)$$

where U_j is the energy of jog formation, c_j is the jog concentration, U_m is the energy of vacancy migration, D_v is the coefficient of vacancy diffusion, U_f is the energy of vacancy formation, c_A is the actual vacancy concentration in the vicinity of the climbing dislocation and is the equilibrium concentration at the temperature T_A, c_o is the equilibrium vacancy concentration at the temperature T, and b is the Burgers vector of the dislocation. Since neither U_j, U_m, or U_f have been measured for gallium, it is not possible to estimate v by direct substitution in Eq. 1. However, it will now be shown that "reasonable" values of U_j, U_m, and U_f can be selected which will give the observed rate of climb.

In the calculations made below it will be assumed that T_A is the melting temperature and that the vacancy concentration present at the melting temperature is retained in the vicinity of the climbing dislocation. Although the actual vacancy concentration is certainly less than the melting temperature concentration, the assumption is valid for the purpose of this disscussion which is to show that the range of values of U_j, U_m, and U_f which are consistent with the observed rate

of loop expansion are not unreasonably large or small in the case of gallium. In fact, the results are essentially unchanged if the number of excess vacancies available to be absorbed by the expanding dislocation loops were only 10% of the value assumed here.

The estimated energy of vacancy formation must satisfy the following criteria 1) there is a reasonable concentration[18] ($\approx 10^{-4}$) of vacancies at the melting temperature and, 2) the number of excess vacancies in the crystal at room temperature must be sufficient to account for the observed dislocation density. Energies of vacancy formation of U_f = 0.2, 0.25 and 0.3 ev provide vacancy concentrations of 5×10^{-4}, 10^{-4}, and 10^{-5}, respectively, at the melting temperature of gallium (≈303 °K). If all of the vacancies present at the melting temperature remain in the crystal as it is cooled to room temperature and are absorbed by the climbing loops, the maximum dislocation density possible in the crystal can be calculated. The average radius of the loops was observed to be about 50 microns and room temperature was around 295°K. Using these values in the calculation, the dislocation densities possible in the crystal for energies of vacancy formation of U_f = 0.2, 0.25, and 0.3 ev are 10^6, 10^5 and 4×10^4 lines/cm^2, respectively. Thus a value of the energy of vacancy formation between 0.2 and 0.3 ev satisfies the criteria mentioned above concerning the required concentrations.

Using this estimated value for U_f, values of the energy of jog formation, U_j, and the energy of vacancy migration, U_m, can now be selected which are consistent with the observed rate of climb. Two cases are considered. First, the value of U_j is assumed to be zero. This represents the case where the jog concentration is not the limiting factor in the rate of climb and results in the

maximum rate of growth for any given value of U_m. The dependence of the velocity of climb, v, on U_m and U_f with $U_j = 0$ is shown by the solid lines of Fig. 3. The sum of the formation and migration energies for vacancies, $U_f + U_m$ is constant on each of the lines and is shown for $U_f + U_m = 0.7$, 0.8, and 0.9 ev. The logarithm of the velocity (in cm/sec) is the ordinate and the ratio $U_f/(U_f + U_m)$ is the abscissa. It is apparent that the velocity is relatively insensitive to the ratio $U_f/(U_f + U_m)$. A value of 0.9 ev for $U_f + U_m$ gives a climb velocity of 10^{-8} cm/sec, the observed rate of growth. If U_f is between 0.2 and 0.3 ev, then the activation energy for vacancy migration necessary to account for the observed rate of climb is 0.6-0.7 ev.

However, if the energy of jog formation is assumed to have a non-zero value, then the required value of U_m is lowered. The dashed lines in Fig. 3 are plotted for the case when U_j is taken to be one-half of the energy of vacancy formation. In contrast to the case when U_j was assumed to be zero, the velocity is strongly dependent on the ratio $U_f/(U_f + U_m)$. The value of $U_f + U_m$ which gives a velocity of 10^{-8} cm/sec is 0.7 ev and the value of U_m is between 0.4 and 0.5 ev, if $U_f = 0.2$ - 0.3 ev. The conclusion that can be drawn is that values of the energies of vacancy formation and migration of U_f in the range 0.2 to 0.3 ev and U_m in the range 0.4 to 0.7 ev are consistent with the observed rate of growth of the loops and the final dislocation density.

This is not to suggest that these estimates of U_f and U_m are to be taken as definitive values, but rather that they are quite conceivable in the case of gallium and thus are consistent with the idea that the expanding dislocation loops are growing by a climb process. No values of U_f, U_m, or $U_f + U_m$ (the activation energy for self-diffusion if one assumes a vacancy

mechanism) determined from other experiments have been reported, therefore no comparisons can be made.

One independent estimate of the value of $U_f + U_m$ can be obtained by using the following empirical equations reviewed by Lazarus.[19] If the relation

$$U_f + U_m = 1.4 \times 10^{-3} T_M \qquad (2)$$

is used (where T_M is the melting temperature in °K), then $U_f + U_m = 0.42$ ev. On the other hand, the equation

$$U_f + U_m = 7.2 \times 10^{-4} L_M , \qquad (3)$$

where L_M is the latent heat of melting in Cal/g-atom, gives a value of $U_f + U_m = 0.96$ ev. The reliability of these estimates is, of course, questionable, but at least the values of $U_f + U_m$ in the range 0.7 to 0.9 ev selected in the preceding discussion lie between the two values given by the empirical equations.

In addition to the observation of the appearance and subsequent growth of dislocation loops in regions initially free of detectable dislocations, the change of initially straight dislocation lines into a "sawtooth" configuration has been observed. The topograph in Fig. 4a, taken 2 days after growth, shows several straight dislocation lines in another part of the crystal shown in Fig. 2. The change to a jagged line over a period of 30 days is shown in Fig. 4b. If the straight dislocation line is a screw dislocation, then the change to a jagged line can be explained in terms of

the development of an irregular helical dislocation by the addition of vacancies to the screw dislocation. The stereo topographs indicate that the orientation of the helical dislocations is consistent with their developing from screw dislocations lying along [011]. The possibility of the occurrence of screw dislocations in this orientation in gallium is consistent with slip in the observed slip system[20] (011) [0$\bar{1}$1] which can be explained in terms of the motion of screw dislocations lying along [0$\bar{1}$1].

The helices exhibit an anisotropic growth behavior similar to that shown by the loops. Both the loops and the helices have a strong preference for expansion in the vertical direction on the topograph. If the development of the loops and helices is attributed to a climb process, then the observed anisotropy in growth indicates that the coefficient of self-diffusion in gallium may be anisotropic. The [100] and [001] axes lie in a vertical plane perpendicular to the plane of the topographs and are inclined 45° to the plane of the topographs so that it is not possible to determine along which of these two directions the diffusion coefficient is greater. However, since the [010] direction lies horizontally in the plane of the topographs, it is possible to observe that the diffusion coefficient in the [010] direction is likely to be the smallest of the three (or at least smaller than one of the other two coefficients), assuming that the plane of the loops is nearly parallel to the plane of the topograph, in accordance with the observations made in the stereo topographs (see above). Thus, the anisotropic growth of the loops indicates that movement of atoms in the (020) planes is favored over motion perpendicular to the planes.

Summary and Conclusions

The observations of this study can be summarized as follows:

1) Dislocation loops have been observed to develop in regions initially free of detectable dislocations and to grow into tangled networks.

2) It is emphasized that the networks develop throughout the bulk of the crystal, many days after the crystal has been grown, and in the absence of any external stress on the crystal.

3) The condition required for the development of the networks is the existence of spatial fluctuations in impurity content in the crystal. It is not known whether this condition controls the nucleation of the loops or the growth of loops which are nucleated in all crystals.

4) The rate of growth of the loops was measured to be 10^{-8} cm/sec.

5) The observed rate of growth and final dislocation density are consistent with a vacancy climb mechanism that requires an energy of vacancy formation of U_f in the range 0.2 to 0.3 ev and an energy of vacancy migration U_m in the range 0.4 to 0.7 ev.

6) The change of initially straight dislocation lines into jagged lines has been observed and is interpreted as the climb of screw dislocations into irregular helical dislocations.

7) The observed anisotropy in the growth of the loops and helices is attributed to an anisotropic coefficient of self-diffusion.

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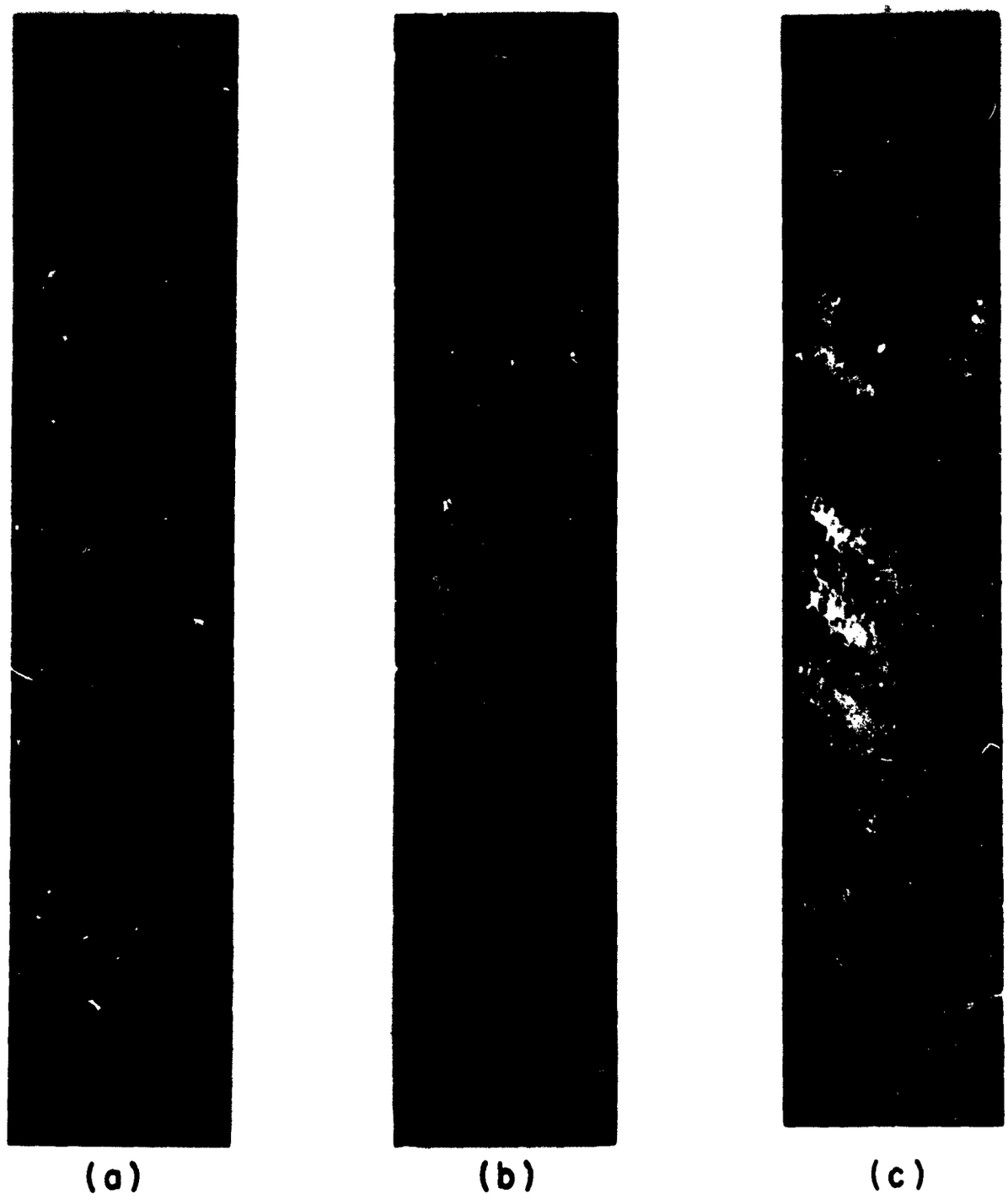
FIGURE CAPTIONS

Fig. 1. Loop growth and network development in a "banded" crystal. (a) Crystal 1 day after growth. (b) Crystal 12 days after growth. (c) Crystal 20 days after growth. The topograph width is about 1.3 mm. The crystal has been rotated 180° in (b) and (c) from its position in (a).

Fig. 2. Growth of dislocation loops. The crystal is shown at (a) 2, (b) 6, (c) 13, (d) 19, (e) 27, and (f) 32 days after growth. The dark spots (particularly noticeable in (b)) are dust particles on the film. The topograph width is about 1.4 mm.

Fig. 3. Velocity of climb of a dislocation as a function of $U_f/(U_f + U_m)$ for different values of $U_f + U_m$ and U_j, where U_f is the energy of vacancy formation, U_m is the energy of vacancy migration, and U_j is the energy of jog formation.

Fig. 4. Development of straight dislocation lines into jagged lines, interpreted as the climb of screw dislocations into irregular helices. Crystal is shown at (a) 2 and (b) 32 days after growth. Lines are in another part of the topographs shown in Figs. 2a and 2f. The topograph width is about 1.4 mm.



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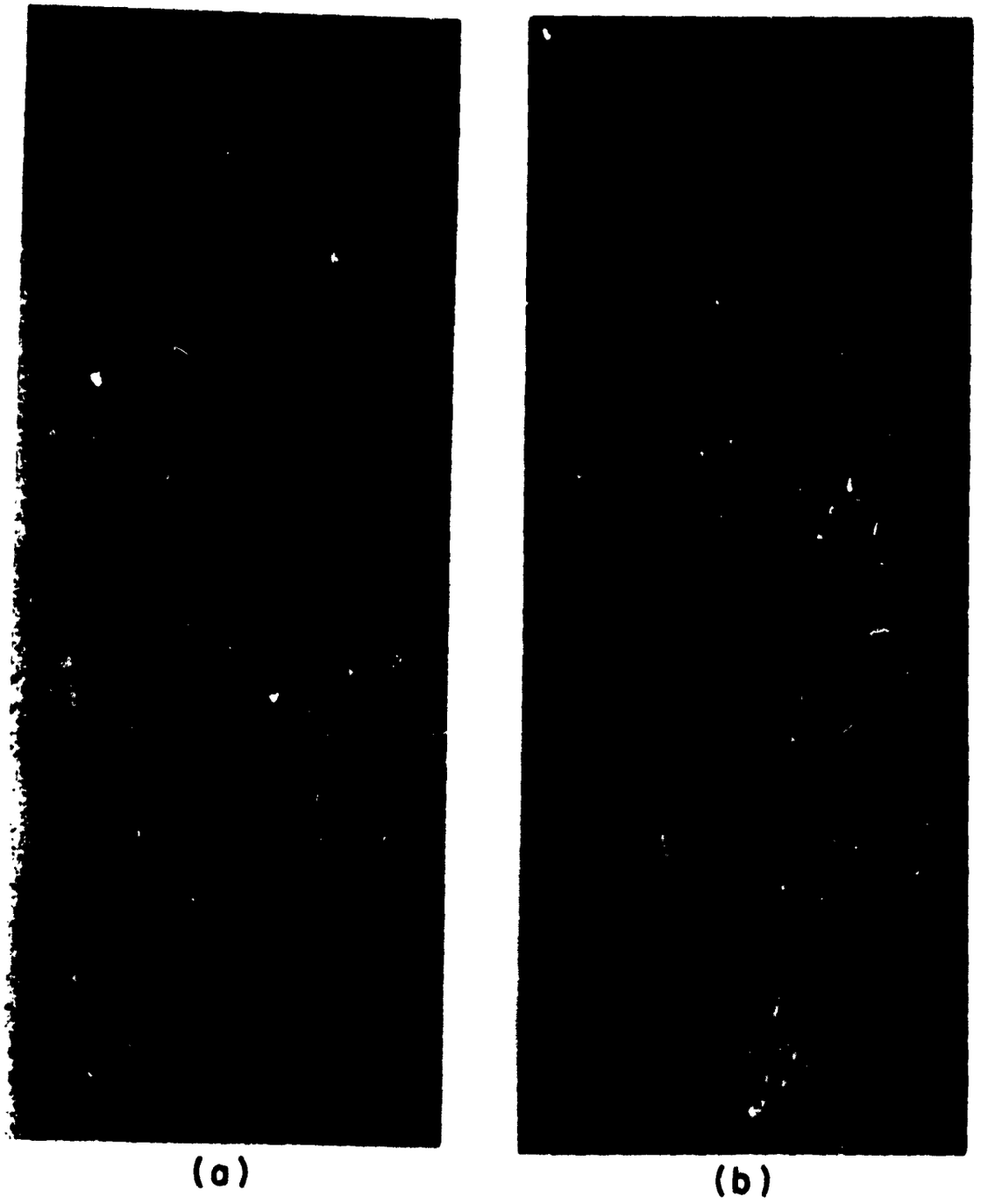


Fig. 2. Growth of dislocation loops. (a) Crystal 2 days after growth. (b) Crystal 6 days after growth. The dark spots (particularly noticeable in (b)) are dust particles on the film. The topograph width is about 1.4 mm.

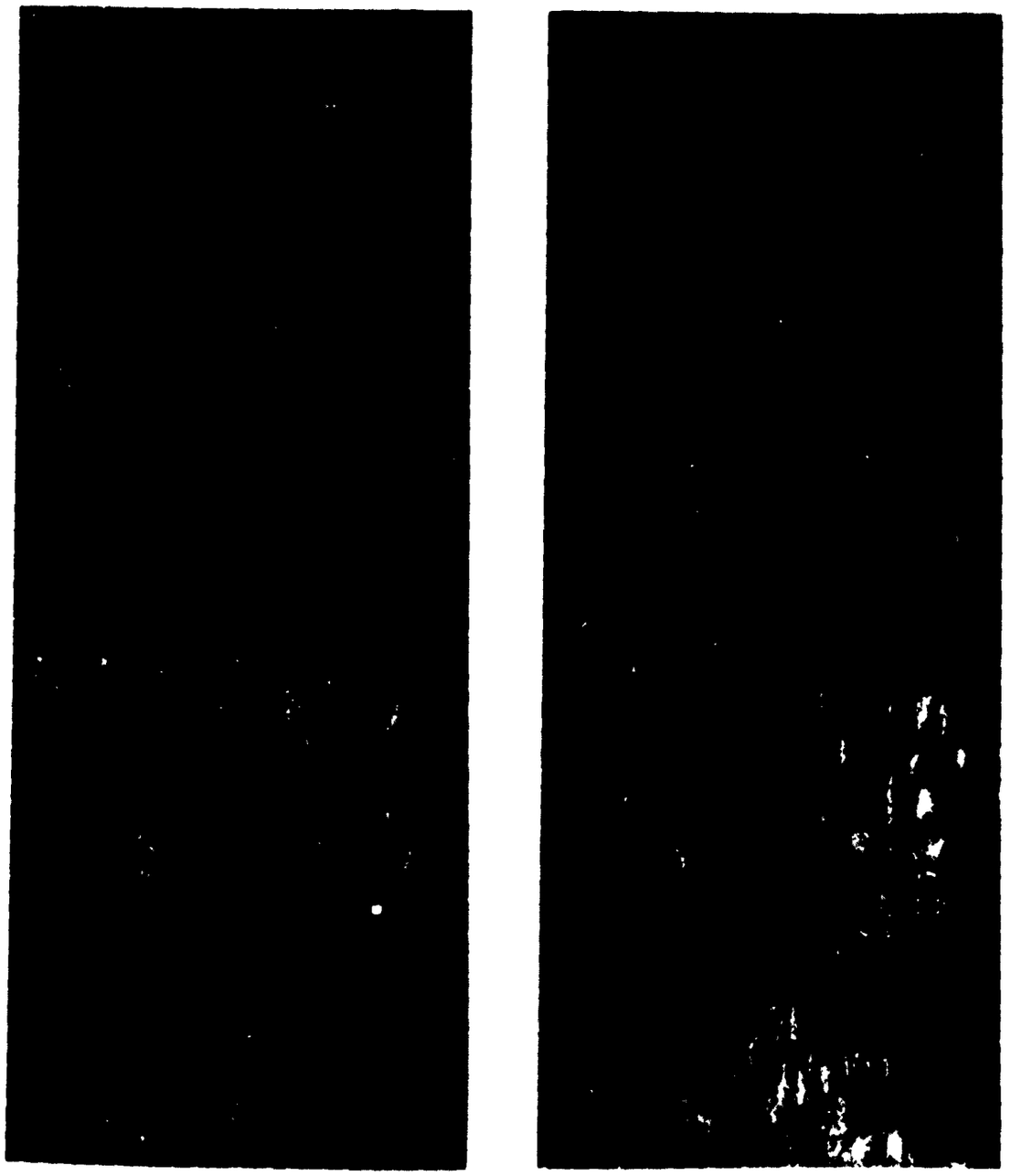


Fig. 2. (con't) (c) Crystal 13 days after growth. (d) Crysral 19 days after growth.

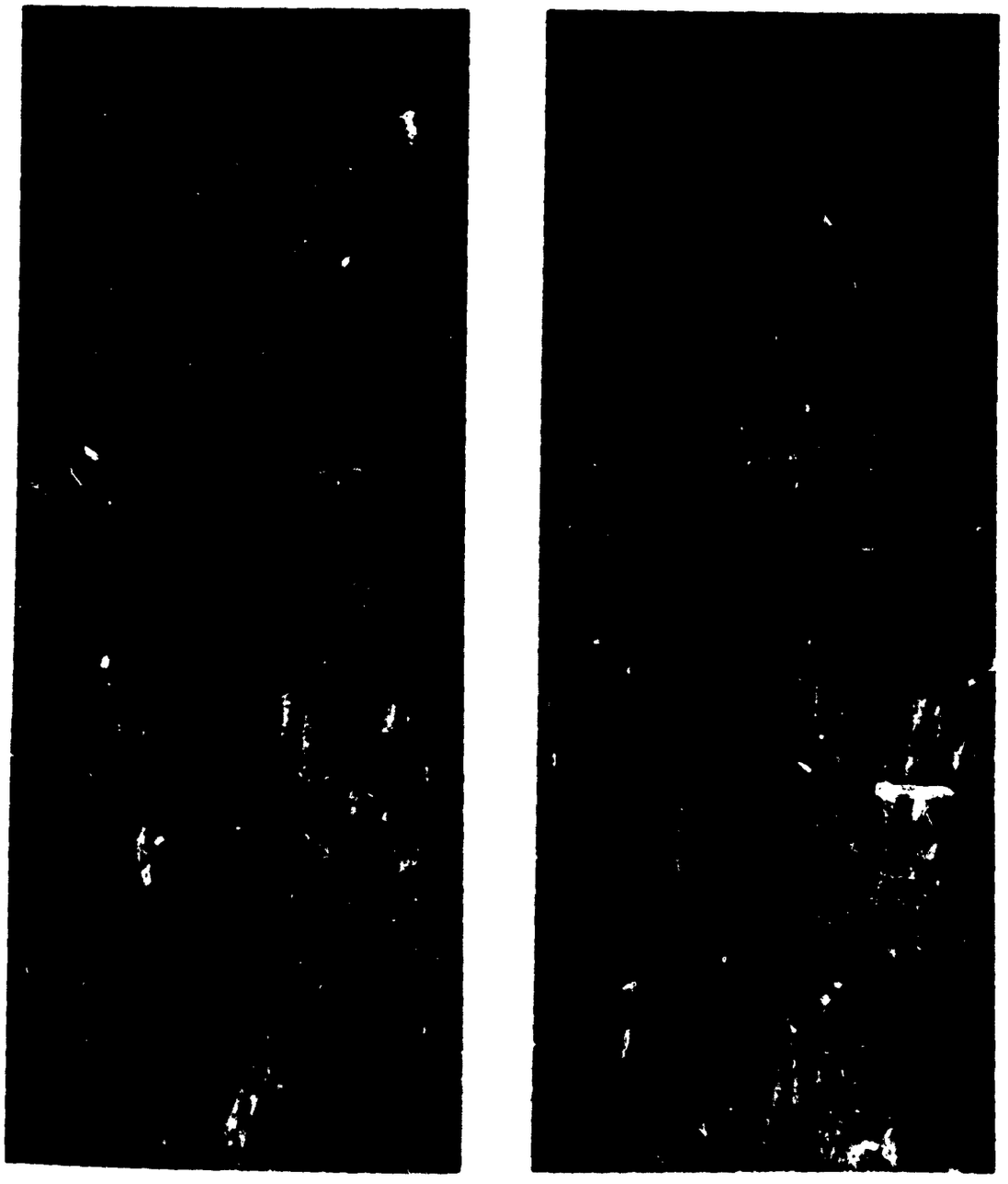


Fig. 2. (con't) (c) Crystal 13 days after growth. (d) Crystal 16 days after growth.

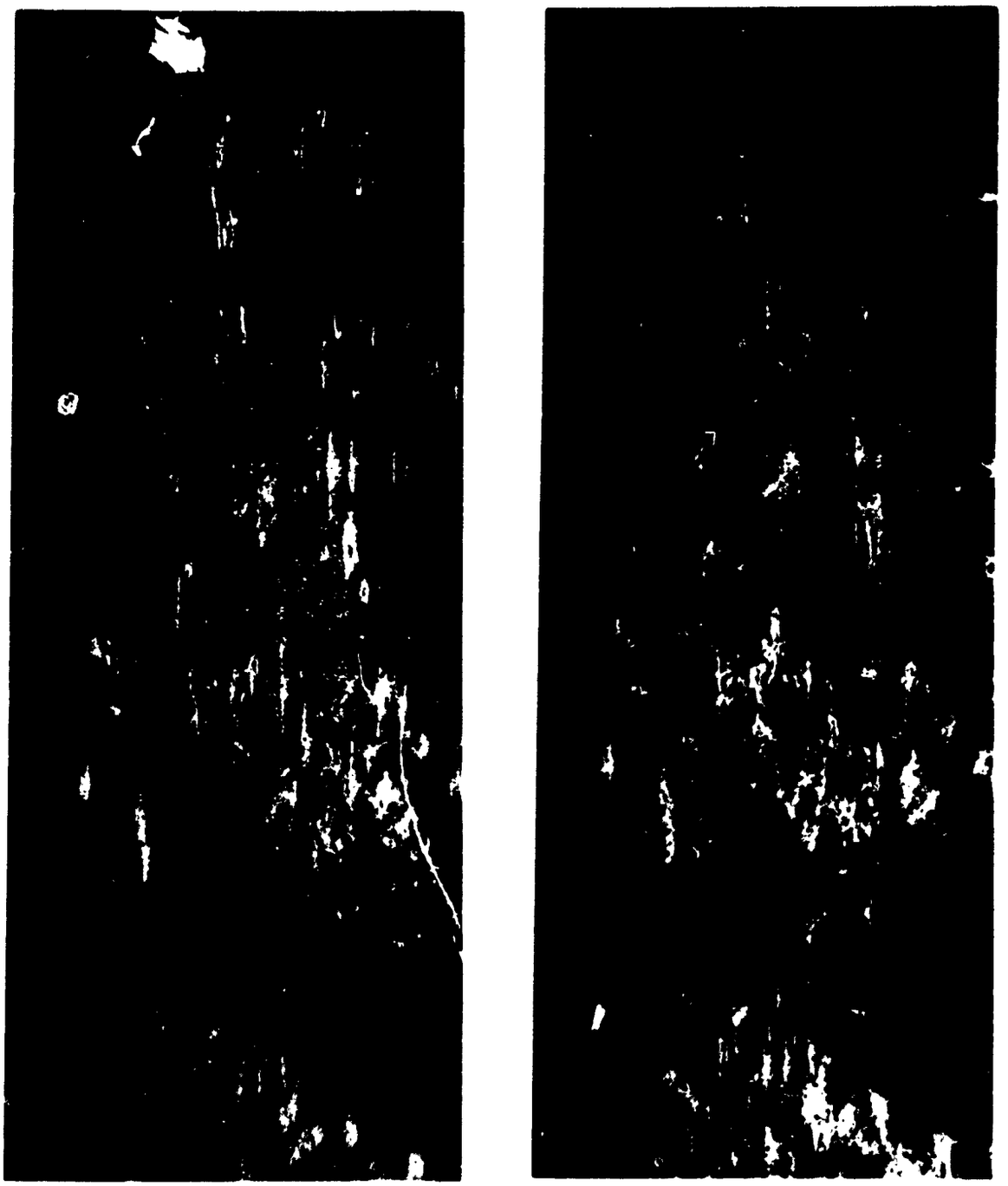
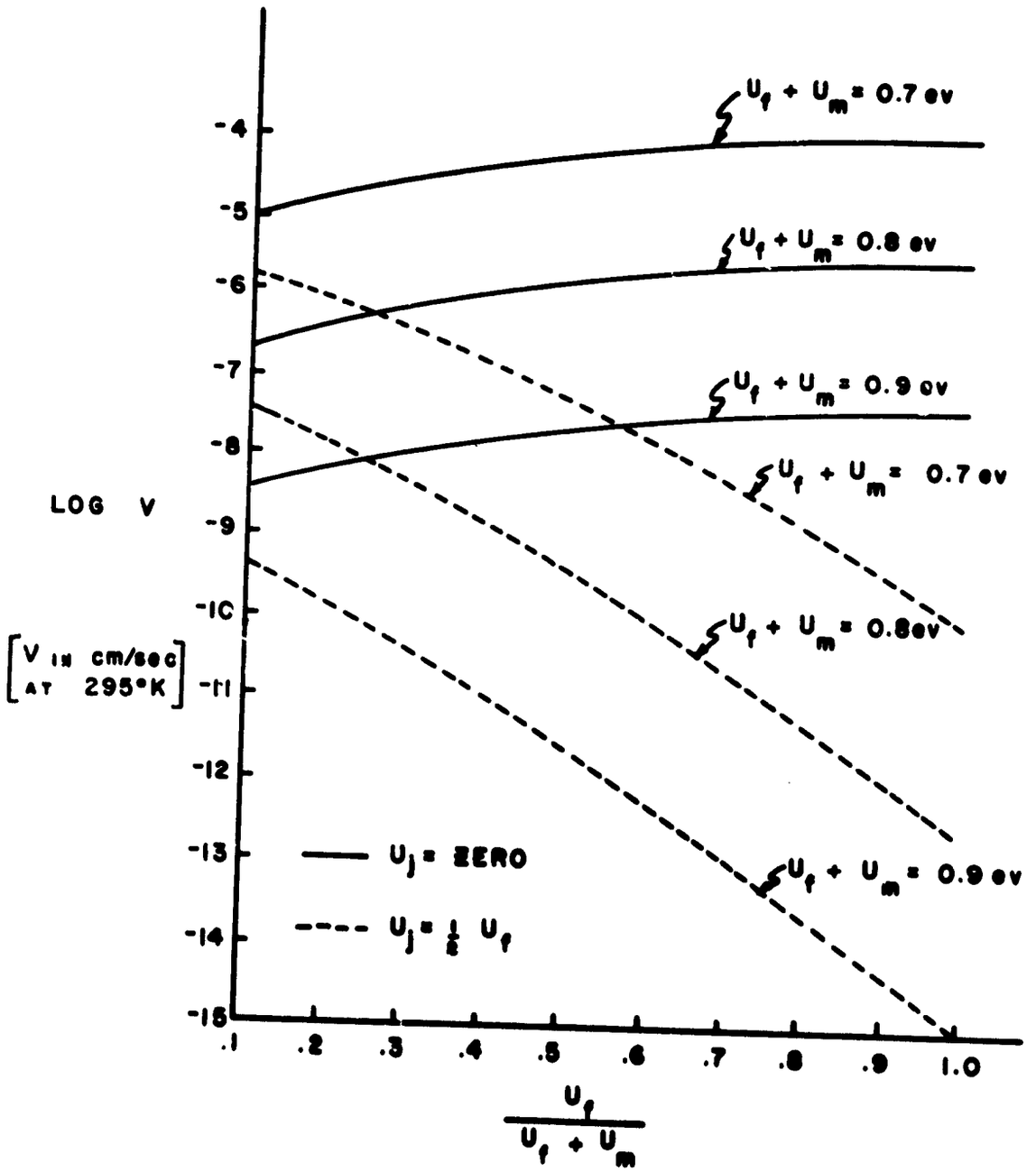


Fig. 2. (con't) (e) Crystal 27 days after growth. (f) Crystal 32 days afte growth



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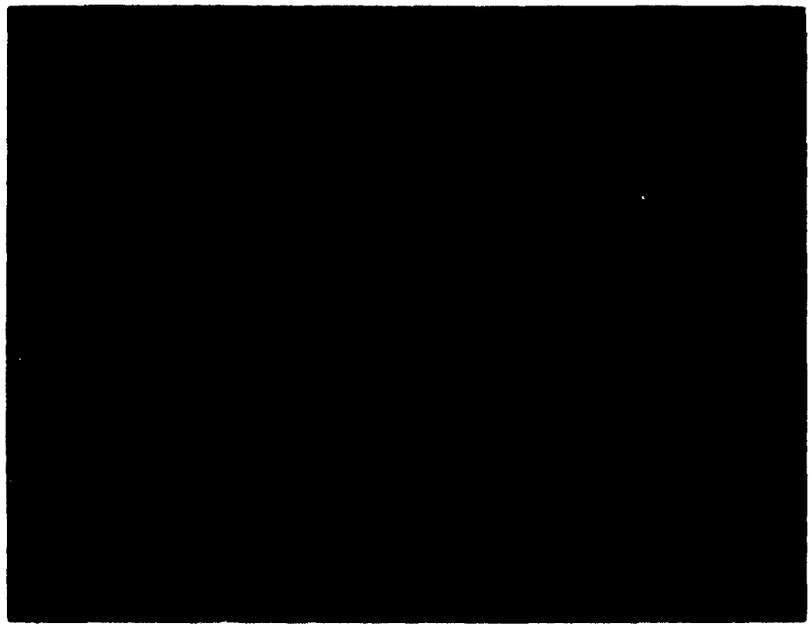
(a)



(b)

Fig. 4. Development of straight dislocation lines into jagged lines, interpreted as the climb of screw dislocations into irregular helices. Crystal is shown at (a) 2 and (b) 32 days after growth. Lines are in another part of the topographs shown in Figs. 2a and 2f. The topograph width is about 1.4 mm.

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13. ABSTRACT

The development of dislocation networks in gallium crystals, initially free of detectable dislocations, has been studied at room temperature by an x-ray diffraction topographic technique. The condition required for the development of these networks is the existence of spatial periodic fluctuations in impurity content. The networks develop, in the absence of an external stress, from individual loops which are first detected 3 to 10 days after the crystal has been grown. These loops grow for days at a uniform rate of 10^{-6} cm/sec and reach a size of a few hundred microns. No specific nucleation mechanism for these loops has been identified. If it is assumed that the loops expand by climb (by the addition of vacancies), their rate of growth and observed densities are consistent with a formation energy for vacancies U_f in the range 0.2 to 0.3 ev and a migration energy for vacancies U_m in the range 0.4 to 0.7 ev. No values of U_f, U_m or $U_f + U_m$ determined from other experiments have been reported and a comparison is not possible at the present time. The change of initially straight dislocation lines into jagged lines has been observed and is interpreted as the climb of screw dislocations into irregular helical dislocations. A marked anistropy in the growth of the loops and helices was observed and is attributed to an anisotropic coefficient of self-diffusion.

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Migration of Vacancies						
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Gallium						

INSTRUCTIONS

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